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PATENT 1422-0401P

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

In Re Application of:

Takayuki MITSUYA et al. : Group Art Unit: 1761

Serial No.: 09/423,085 :

Filed: November 2, 1999 : Examiner: MADSEN, Robert

For: POWDER COMPOSITION

DECLARATION UNDER 37 C.F.R. 1.132

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WASHINGTON, D.C. 20231

Sir:

I, Senji SAKANAKA, residing at Mie-ken, Japan, hereby declare and state as follows:

1. That I am one of the co-inventors of U.S. Application Serial No. 09/423,085 filed on November 2, 1999. I am thoroughly familiar with the contents of said Application, its prosecution before the United States Patent and Trademark Office and the references cited therein.

2. That I am a graduate of Graduate School of Osaka Prefecture University, Department of Applied Biochemistry in the year 1976, majoring in fermentation chemistry.

3. That I have been employed in Taiyo Kagaku Co., Ltd. in the year 1977 and have been assigned to the Research Laboratories.

4. That I have been involved in the research and development of functional foods.

5. That the following experiments were conducted by myself or under my direct supervision and control in order to verify that a comparative study of the powder composition of the present invention with the composition of Japanese Patent Laid-Open No. Hei 9-9878 (hereinafter simply referred to as "JP'878") reveals that the powder composition of the present invention is clearly different from the composition of JP'878 and exhibits unexpectedly excellent effects.

METHODS

First, delipidated egg yolk particles (A) were prepared in accordance with the process of JP'878, and delipidated egg yolk particles (B) were prepared in accordance with the process of the present invention to conduct a comparative study on the flowability and the flavor.

PREPARATION OF DELIPIDATED EGG YOLK PARTICLES (A) OF JP'878

The delipidated egg yolk particles (A) were prepared in accordance with the process of Example 1 of JP'878. Specifically, 500 parts by weight of ethanol was added, based on 10 parts by weight of an egg yolk powder, and the mixture was stirred in a homomixer, and filtered with a filter press. The resulting delipidated product was dried with a rotary vacuum dryer, and thereafter classified at 40 mesh, to give delipidated egg yolk particles (A).

PREPARATION OF DELIPIDATED EGG YOLK PARTICLES (B) OF THE PRESENT INVENTION

The delipidated egg yolk particles (B) were prepared in accordance with Preparation Example 1 of the present invention. Specifically, to 100 kg of egg yolk powder was added 2000 liter of ethanol, and the mixture was stirred with a

homomixer at 40°C for 30 minutes. The resulting mixture was filtered with a flat plate-type filtration apparatus using a filter paper. To the resulting filtration residue was added 200 kg of deionized water, and mixed. Thereafter, the mixture was dried with a spray-dryer (manufactured by Ohgawara Kakoki under the trade name of Model "DC16", inlet: 140°C, outlet: 75°C), whereby giving 32 kg of delipidated egg yolk particles which were porous, having a large number of pores on the particle surface. The pores were confirmed with SEM. The pore size was such that the diameter was about 0.1 to about 10 μm. The delipidated egg yolk particles are denoted as (B).

Next, each of the delipidated egg yolk particles (A), (B) was stirred with fish oil into a homogeneous mixture with Super Mixer or under reduced pressure in the manner described below.

PREPARATION OF POWDER COMPOSITION (AS, BS)

To 10 kg of each of the delipidated egg yolk particles (A) and (B) was added 3 kg of a fish oil (DHA content: 25% by weight). The mixture was stirred into a homogeneous mixture with Super Mixer, to give 13 kg of each powder composition (AS, BS). Here, AS denotes the powder composition prepared from the delipidated egg yolk particles (A); and BS denotes the powder composition prepared from the delipidated egg yolk particles (B).

PREPARATION OF POWDER COMPOSITION (AR, BR)

To 10 kg of each of the delipidated egg yolk particles (A) and (B) was added 3 kg of a fish oil (DHA content: 25% by weight). The mixture was stirred into a

homogeneous mixture at 30°C for 1 hour under reduced pressure (30 mmHg) with a vacuum kneader, to give 13 kg of a powder composition (AR, BR). Here, AR denotes the powder composition prepared from the delipidated egg yolk particles (A); and BR denotes the powder composition prepared from the delipidated egg yolk particles (B).

Next, comparative tests for each of powder compositions AS, BS, AR and BR on the flowability (angle of repose) and the flavor were carried out as follows.

COMPARATIVE TESTS

(1) FLOWABILITY

The flowability as evaluated by an angle of repose was determined in the same manner as in Test Example 1 of the present invention. Specifically, an angle of repose for a 500 g sample of each of powder compositions AS, BS, AR and BR was measured by an analyzer for determining an angle of repose by Miwa type cylindrical rotation method (manufactured by Tsutsui Rikagaku Kikai K.K.) under the conditions of a relative humidity of 40% and a temperature of 25°C.

(2) FLAVOR

The flavor as evaluated by fish odor was determined in the same manner as in Test Example 2 except that a sensory evaluation was carried out for fish odor in place of bitterness. Specifically, panelists consisting of 5 each of male and female normal individuals of an age of 24 to 35 years old were subjected to sensory examination for "fish odor" with each of powder compositions AS, BS, AR and BR by placing 20 mg of each powder on the tongue. The evaluation criteria are as follows.

Evaluation Scores

0 No fish odor;

1 Slight fish odor;

2 Little fish odor;

3 Some fish odor;

4 Marked fish odor; and

5 Strong fish odor.

The value obtained by dividing the total sum of the evaluation scores for "fish oil" for each individual panelists by the total number of the panelists (10 persons) was defined as sensory evaluation for "flavor."

RESULTS

The results are shown in Table I.

Table I

Sample	Angle of Repose	Fish Odor
AS	75°	2.9
BS	70°	2.5
AR	65°	2.6
BR	50°	1.1

DISCUSSION

It is clear from Table I that the best result for the angle of repose and the best result for the flavor are obtained when using the powder composition BR of the present invention, prepared by stirring the delipidated egg yolk particles (B) with a fish oil under reduced pressure with a vacuum kneader. We assume that this is because the delipidated egg yolk particles prepared according to the process of the present invention have many pores, into which fish oil is substantially impregnated. Since only very little fish oil would be present on the surface of the resulting particles, if present, making the surface unsticky, the powder composition BR has flowability as shown by a small angle of repose. In addition, since the fish oil is present in the pore, there would be no problem in odor. On the other hand, even when the delipidated egg yolk particles (B), i.e. those particles having pores, were used, the fish oil is less likely to be impregnated by the use of Super Mixer, a substantial amount of the fish oil would remain on the surface of the particles, thereby making its flowability notably poorer in the powder composition BS. Further, when the delipidated egg yolk particles are prepared with a rotary vacuum dryer, not by means of spray-drying, pores are not formed on the surface of the particles, so that the fish oil again remains on the surface of the particles. Therefore, the powder compositions AS and AR have poorer flowability and consequently poorer odor.

Therefore, unexpectedly excellent effects of the present invention can only be obtained by the combined means of spray-drying for the preparation of the delipidated egg yolk particles and a treatment under reduced pressure with a vacuum kneader for enabling impregnation of the fish oil as employed in the present invention.

Accordingly, the present invention is not obvious over JP'878.

6. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

7. Further declarant saith not.

Senji SAKANAKA

May 25, 2001

Date

PARTIAL ENGLISH TRANSLATION of Japanese Patent Laid-Open No. Hei 9-9878

Laid-Open Date: January 14, 1997

Application No. Hei 7-180940

Filing Date: June 23, 1995

Applicant: Taiyo Kagaku Co., Ltd.

[Title of the Invention]

Functional Protein Preparation and Process for Preparing the Same

[Claims]

[Claim 1] A composition having an anti-oxidative ability, comprising a delipidated egg yolk.

[Claim 2] A process for preparing a composition having an anti-oxidative ability, characterized by delipidating an egg yolk.

[Claim 3] A powdery fat or oil, characterized in that the powdery fat or oil comprises the composition having an anti-oxidative ability of claim 1.

Column 2, right column, line 7 - column 3, left column, line 44

[0005]

[Means to Solve the Problems] In order to solve the above problems, therefore, the present inventors have intensively studied on the anti-oxidative stability of fats and oils by using a substance having good oil-absorbing ability. As a result, they have found that a substance having a higher oil-absorbing ability and also having a higher anti-oxidative effect which was not conventionally obtained can

be obtained by delipidating an egg yolk and thereafter subjecting the delipidated egg yolk to a purification treatment. Further, they have found that a composition having an anti-oxidative ability comprising the delipidated egg yolk is mass-producible, so that the composition is inexpensive, and that the composition can be used in a wide range owing to non-stickiness and good tastes and colors. The detailed description of the invention will be given as follows. The present invention is characterized by a composition having an anti-oxidative ability, comprising a delipidated egg yolk having a high oil-absorbing ability and an anti-oxidative ability and a process for preparing the same, and a powdery fat or oil having a high stability, which was not conventionally obtained, by mixing the composition with a fat or oil. The phrase "composition having an anti-oxidative ability, comprising a delipidated egg yolk" in the present invention refers to a composition obtained by purifying a delipidated egg yolk prepared by removing an oily component from an egg yolk, and drying the delipidated egg yolk. The term "delipidated egg yolk" in the present invention refers to one obtained by drying a product prepared by removing a lipid from an egg yolk. As to the removal of the oily component referred to herein, the lipid content is not limited, and the content is desirably 10% or less, more desirably 1% or less, most desirably completely removing the oily component. By the removal of the oily component, an oily component which is intended to be stabilized can be contained in a large amount during the preparation of the powdery fat or oil, so that the stickiness caused by an increase in the oily component can be prevented.

[0006] The egg yolk used as a raw material may be a raw egg or an egg yolk powder. The delipidation may be carried out with a solvent generally used in the manufacturing of foods, concretely including ethanol, acetone, hexane, and the

like, and ethanol being desired. The amount of the solvent is not particularly limited, and it is preferable that the solvent may be used in an amount of 100 to 200 parts, based on 10 parts of the egg yolk. In addition, the purification treatment after the delipidation in the present invention means that the egg yolk is washed by adding water after delipidation. More specifically, to the delipidated egg yolk after the delipidation treatment is added water in about a 5-fold quantity or more, and the mixture is sufficiently stirred, and filtered, and the solid ingredient is dried. As to the filtration in this treatment, there can be employed filter press, or compression filtration. Also, the drying method during this treatment is not particularly limited, and spray-drying or the like may be preferably carried out. By the drying process, the anti-oxidative ability is further enhanced. In addition, the fats and oils in the present invention may be any of those which can be generally used for foods, such as soybean oil, rapeseed oil, fish oil, beef tallow, lard, nut oil, sesame oil, corn oil, safflower oil and milk fat. Further, the fat or oil may contain PUFA which is more susceptible to oxidation such as DHA, EPA or γ-linolenic acid, and its content is not limited. Also, the mixing as referred to herein may be simply a step of homogeneous stirring with Super-Mixer or the like, without necessitating dispersing with water or the like or spray-drying. Therefore, the time required for the preparation of the powdery fat or oil is shortened, so that the risks of the deterioration and bacterial contamination of the fat or oil can be reduced.

[0007] Next, the powdery fat or oil obtained according to the present invention may be directly edible, or it can be used for beverages, confectioneries, and a wide variety of food materials. Although the texture may depend upon the content of the oil, the powdery fat or oil obtained in the present invention has a

remarkably smooth texture, as compared the conventional powdery fat or oil with stickness and oiliness, thereby broadening the applications due to its tastes, handling, and easiness in processing. Further, other food materials such as proteins, sugars, vitamins and minerals can be formulated with the fat or oil of the present invention, and processed. Next, the following examples are given, without intending to restrict the scope of the present invention thereto.

[0008]

[Examples]

Example 1

Five-hundred parts by weight of ethanol was added, based on 10 parts by weight of an egg yolk powder, and the mixture was stirred in a homomixer, and filtered with a filter press. The resulting delipidated product was dried with a rotary vacuum dryer, and thereafter classified at 40 mesh, to give a composition having an anti-oxidative ability.

Example 2

Six-hundred grams of a fish oil (trade name: DHA-27, manufactured by Tama Seikagaku K.K.) was added to 3 kg of the composition having an anti-oxidative ability prepared in Example 1, and the mixture was stirred with Super-Mixer into a homogeneous state, to give a powdery fat or oil. The peroxidant value (hereinafter simply referred to as "POV") in this case was 0.6 meq/kg. The powdery fat or oil was stored in a thermostat kept at 60°C, and sampled with the passage of time to determine the POV. The results are shown in Figure 1.

[0009] Example 3

One kilogram of a fish oil (trade name: DHA-27, manufactured by Tama Seikagaku K.K.) was added to 3 kg of the composition having an anti-oxidative

ability prepared in Example 1, and the mixture was stirred into a homogeneous state, to give a powdery fat or oil. The POV in this case was 0.6 meq/kg. The powdery fat or oil was stored in a thermostat kept at 60°C, and sampled with the passage of time to determine the POV. The results are shown in Figure 1.

Example 4

The amount 1.5 kg of a fish oil (trade name: DHA-27, manufactured by Tama Seikagaku K.K.) was added to 3 kg of the composition having an anti-oxidative ability prepared in Example 1, and the mixture was stirred into a homogeneous state, to give a powdery fat or oil. The POV in this case was 0.6 meq/kg. The powdery fat or oil was stored in a thermostat kept at 60°C, and sampled with the passage of time to determine the POV. The results are shown in Figure 1.